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Preparation of a series of N-alkyl-3-boronopyridinium halides and study of their stability in the presence of hydrogen peroxide

Research Article

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Abstract: A simple and efficient protocol for the preparation of a series of N-alkyl-3-boronopyridinium salts is described which requires exposure of 3-pyridineboronic acid neopentylglycol ester and corresponding alkyle halide to microwave irradiation followed by boronic ester hydrolysis. The technique employed drastically reduces the reaction time and prevents thermal degradation and the formation of side products. Water solutions of the obtained boronopyridinium salts are shown to be stable at room temperature in wide pH range as well as in the presence of hydrogen peroxide at pH 10.0 for 72 h.

Keywords: *3-Pyridineboronic acid • Green chemistry • Alkylation • Microwave activation • Peroxide activation*

1. Introduction

The interest of chemists in boronic acids has risen due to their unique properties such as mild organic Lewis acids and acting as reactants under the mild condition, coupled with their ease of handling [1]. All these properties make boronic acids a particularly attractive class of synthetic intermediates [2], scaffolds for functional materials [3] and self-assembly [4], and molecular blocks for saccharide recognition [5]. Because of the relatively low toxicity of boronic acids and their degradation into the environmentally benign boric acid they can be considered "green" compounds. Yamamoto and coworkers recently tested several reusable *N*-alkylated pyridineboronic acid catalysts for the dehydrative amide condensation between equimolar mixtures of acids and amines [6,7]. Boronic acids combined with hydrogen peroxide can be a source of peroxoboronate, a species of higher oxidizing power than that of initial hydrogen peroxide, as it replaces peroxoborates [8] or oxone [9]. Surfactants functionalized with a boronate moiety were shown recently [10] to be effective coupling reagents. We are interested in exploring the possibility of using *N*-alkylated pyridine-3-boronic acids for the design of "mild" oxidative microheterogeneous systems [11], polyol-recognition systems, and in the preparation of *N*-alkylpyridinium aryl ketones. Despite the high synthetic value of 4-pyridine boronic acid derivatives, [7] it should be noted that they are much less soluble in water and will thus be of less importance as the precursors of micellar oxidative systems.

Recently, we have reported a simple method of preparation of *N*-alkyl-3-boronopyridinium triflates [12], in which the general method for the synthesis of N-alkyl boronopyridinium halides involves the condensation of 3-pyridylboronic acid and an alkylating agent with nitromethane as the solvent under reflux for 3, 7, and 12 days [13].

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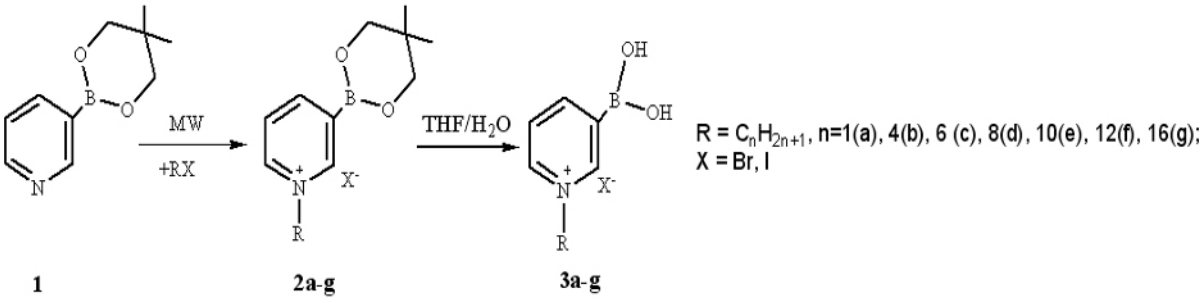

Figure 1. Scheme for *N*-alkyl-3-boronopyridinium halide preparation using microwave irradiation.

Among the several aspects of green chemistry, the reduction/replacement of volatile organic solvents from the reaction medium is of utmost importance. The use of a large excess of conventional volatile solvents required to conduct a chemical reaction creates ecological and economic concerns [14]. Organic reactions accelerated under the influence of microwave (MW) irradiation have attracted considerable attention in past decades for the efficient and relatively friendlier synthesis of a variety of organic compounds [15-18]. In recent years, microwave irradiation using both laboratory monomode and commercially available multimode ovens is increasingly used for the optimization and acceleration of organic synthesis under solvent free conditions [18-20]. Previously it was reported the synthesis of *N*-alkylated pyridinium boronic acids under microwave irradiations [21], starting from 3-alkoxy-2-bromopyridines.

We report herein an efficient method for the preparation of *N*-alkyl-3-boronopyridinium halides using microwave irradiation as energy source by simple exposure of reactants to microwave irradiation under solvent free conditions, and studies of the stability of the boronopyridinium iodides towards degradation in the presence of hydrogen peroxide in water.

2. Experimental procedure

All chemicals were purchased as reagent grade from Aldrich and used without further purification, unless otherwise noted. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AVANCE 300 MHz and Bruker AVANCE 500 MHz instruments. ^{11}B spectra were recorded in quartz tubes on AVANCE 400 MHz instrument. The ESI-MS were obtained from a Q-Tof Ultima (Waters). A multimode microwave oven (Samsung GE107W; microwave frequency 2450 MHz) was used for carrying out the reactions. UV-Vis spectra recorded using HP 8452A Diode Array spectrophotometer with thermostated cell unit.

Each experiment on synthesis was performed by both the classical reflux method and in the microwave oven. In each case, the same quantities of reagents were used and the products were worked up and purified in the same way. Each reaction was run several times to find the reaction times that gave the same yields by the conventional and microwave technique. This allowed the relative rate enhancement for each experiment to be estimated.

In a typical reaction, alkyl halide (1.25 mmol) and **1** (1 mmol) were mixed thoroughly, placed in an open pyrex container and the reaction mixture irradiated with microwave oven at 300 W (30 s irradiation followed by mixing) until yellowish (iodide) or brownish (bromide) color was obtained. The progress of the reaction was monitored by ^{1}H NMR. The solid obtained was washed with dry ether to remove unreacted starting materials. The resulting yellowish solid was added to a mixture of THF/ H_2O, and the solution was stirred at room temperature. The solvent was removed *in vacuo*, and the aqueous solution was washed with ether until neopentyl glycol was completely extracted. The water was removed by freeze drying and the resulting yellow solid was washed thoroughly with dry ether and recrystallized from methanol/ether. Alternatively, for obtaining **3f** and **3g** with good yield, the hydrolysis technique was modified by using a mixture of dichloromethane and water sodium bicarbonate (9:1). The pH of the water phase was maintained at 9.5. After stirring for 12 h under ambient conitions, the organic phase was decanted and the water layer was washed with an excess of dichlomethane. Organic solvent was removed *in vacuo*, and final product was recrystallized from methanol/ether.

Typical spectra of the compouds obtained are given below.

N-methyl-3-boronopyridinium iodide neopentyl ester **(2a).** ^{1}H NMR (300 MHz, DMSO-d_6) δ_H 1.00 (s, 6H, C(CH_3)$_2$), 3.82 (s, 4H, OCH_2), 4.64 (s, 3H,

CH_3); 8.10 (t, 1 H, J= 6.5 Hz, Pyr-H5), 8.64 (d, 1H, J=6.5 Hz, Pyr-H6), 9.00 (d, 1H, J=6.5 Hz, Pyr-H6), 9.04 (s, 1H, Pyr-H2); ^{13}C NMR (75 MHz, DMSO-d_6) δ_C 21.1 (C(CH_3)$_2$), 31.5 (q C), 47.6 (Me), 71.5 (OCH_2), 127.1 (Pyr-C5), 146.5 (Pyr-C6), 148.7 (Pyr-C4), 148.9 (Pyr-C2); HR ESI MS m/z calcd for $C_{11}H_{17}BNO_2$ (M-I) 206.1347, found 206.1337.

N-dodecyl-3-boronopyridinium iodide neopentyl ester **(2f)**. ^{1}H NMR (300 MHz, DMSO-d_6) δ_H 0.87 (t, 3H, J=6.5 Hz, CH_3), 1.00 (s, 6H, C(CH_3)$_2$), 1.22-1.36 (br s, 18H, 9CH_2), 1.95-2.05 (m, 2H, NCH$_2$CH_2), 3.84 (s, 4H, OCH_2), 4.64 (t, 2H, J=7 Hz, NCH_2); 8.13 (t, 1 H, J= 6.5 Hz, Pyr-H5), 8.68 (d, 1H, J=7 Hz, Pyr-H6), 9.11 (d, 1H, J=6.5 Hz, Pyr-H4), 9.14 (s, 1H, Pyr-H2); ^{13}C NMR (75 MHz, DMSO-d_6) δ_C 13.9 (Me), 21.2 (C(CH_3)$_2$), 22.0 (CH_3CH_2), 25.3 ($CH_3CH_2CH_2$), 28.2, 28.6, 28.7, 28.8, 28.9, (dodecyl-CH_2), 30.8 (NCH$_2$CH$_2$CH$_2$), 31.2 (NCH$_2$CH$_2$), 31.5 (q C), 60.4 (NCH_2), 71.5 (OCH_2), 127.4 (Pyr-C5), 145.6 (Pyr-C6), 148.1 (Pyr-C4), 149.1 (Pyr-C2); HR ESI MS m/z calcd for $C_{22}H_{39}BNO_2$ (M-I) 360.3068, found 360.2850.

N-methyl-3-boronopyridinium iodide **(3a)**. ^{1}H NMR (300 MHz, CD_3OD) δ_H 4.42 (s, 3H, CH_3), 8.05 (t, 1 H, J= 7 Hz, Pyr-H5), 8.73 (d, 1H, J=7 Hz, Pyr-H4), 8.87 (d, 1H, J=6.5 Hz, Pyr-H6), 8.95 (s, 1H, Pyr-H2); ^{13}C NMR (125 MHz, CD_3OD) δ_C 49.5 (Me), 128.5 (Pyr-C5), 147.4 (Pyr-C6), 150.8 (Pyr-C4), 152.3 (Pyr-C2); HR ESI MS m/z calcd for $C_6H_7BNO_2$ (M-I) 138.0721, found 137.0687

N-dodecyl-3-boronopyridinium iodide **(3f)**. ^{1}H NMR (300 MHz, CD_3OD) δ_H 0.90 (s, 3H, CH_3), 1.24-1.38 (br s, 18H, 9CH_2), 1.91-1.98 (m, 2H, NCH$_2$CH_2), 4.55 (t, 2H, J=7 Hz, NCH$_2$CH_2); 7.90 (t, 1 H, J= 6.5 Hz, Pyr-H5), 8.56 (d, 1H, J=7 Hz, Pyr-H4), 8.68 (s, 1H, Pyr-H6), 8.70 (d, 1H, J=6.5 Hz, Pyr-H2); ^{13}C NMR (125 MHz, CD_3OD) δ_C 14.5 (Me), 23.8 (CH_3CH_2), 27.2 ($CH_3CH_2CH_2$), 30.1, 30.2, 30.5, 30.6, 30.6, 30.7 (dodecyl-CH_2), 30.8 (NCH$_2$CH$_2$), 62.5 (NCH_2), 127.8 (Pyr-C5), 143.2 (Pyr-C6), 148.6 (Pyr-C4), 150.9 (Pyr-C2); HR ESI MS m/z calcd for $C_{17}H_{37}BNO_2$ (M-I) 292.2442, found 292.2291

3. Results and discussion

We examined the effect of microwave irradiation and organic solvent additives on a set of the reactions of alkylhalides with 3-pyridine boronic acid (PBA) and 3-pyridineboronic acid neopentylglycol ester **1** (Fig. 1).

Direct alkylation of PBA with RX under MW irradiation of moderate power in the presence and absence of an organic solvent leads to the formation of stable adducts, which strongly depends on the alkyl halide chainlength. Under these conditions, PBA is supposed to form the partially alkylated adduct with stoichiometry from 1:4 (R=CH_3) to 1:1 (R=$C_{12}H_{25}$, $C_{16}H_{33}$). Boronic acids are well known to form acyclic adducts with bases [22] or undergo dehydration to form anhydrides; some of these anhydrides are stable (an excellent example is cyclotrimeric anhydrides boroxines [23] being of interest as ring structures incorporated into functional materials and macromolecular architectures [3]). However, there were no peaks found in the ESI-MS spectra corresponding to the primary molecular ion of the adducts of the supposed structure, probably due to the fast decomposition under the electron spray condition.

UV study of the adduct of 3-PBA and methyl iodide in water demonstrates no changes in the pH range between pH 6 and 10 (Fig. 2a) whereas in the presence of the excess of H_2O_2 the remarkable changes appear with increasing of pH (Fig. 2b) which is not shown for *N*-methyl-3boronopyridinum iodide **3a** at pH providing complete transformation into tetrahedral boron. This may be ascribed to the ionic equilibria with an adduct as well as with products of its cleavage.

An experiment using ^{1}H NMR and 2D NMR techniques (HMBC, HSQC, and ^{1}H-^{1}H COSY NMR; CD_3OD) allows us to confirm the formation of adducts whose structure will be clarified in a separate work. On increasing the microwave power level, degradation (deboronation) products appear, suggesting that these conditions are too harsh. Thus, target product (type **2)** cannot be obtained under these conditions.

A protecting group on the boron moiety (pyridine boronic acid neopentyl ester **1**) prevents the risk of formation of substantial by-product amounts and make it possible to complete the alkylation. Upon microwave irradiation, the *N*-alkyl-3-boronopyridinium ester halide **2a-g** appears that increases the polarity of the reaction medium thereby increasing microwave absorption. The formation products **2a-g** can be monitored visibly in the reaction when it turns from a clear solution to yellow (iodide) or brownish (bromide).

Typical procedure includes reaction of **1** with dodecyl iodide. It is observed that, at elevated power levels, evaporation of alkyl halide and partial decomposition/charring of the salt occurs, which eventually results in lower yields. To circumvent this problem, we conducted the reaction with intermittent heating and mixing at a moderate power level to provide a better yield and cleaner salt formation. After

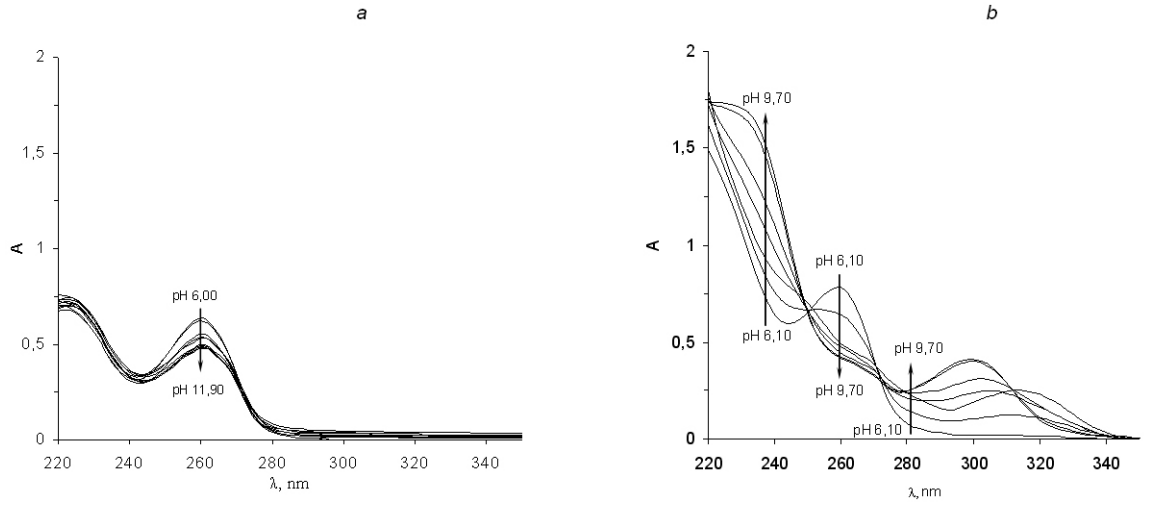

Figure 2. UV spectra of the adduct of 3-PBA (22 mg/l) with CH_3I at different pH in water (a) and in the presence of 0.5 mM H_2O_2 (b); 1 M KCl; water, 25°C.

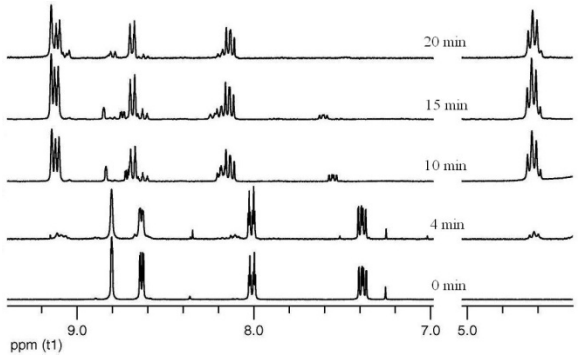

Figure 3. ^{1}H NMR (DMSO-d_6) spectra for the reaction of **1** with iodododecane at different times of microwave irradiation (300 W).

Table 1. Optimization of reaction conditions for microwave-assisted preparation of 12f iodide; Loading: 1.25 mmol $C_{12}H_{25}I$; 1.0 mmol 1.

N°	MW Power, W	Time, min	Yield, %
1	180	5	<5
2	300	9	10
3	300	25	45
4	300	40	90
5[a]	450	5	56
6[b]	300	4	10
7	300	10	80
8[b]	300	15	88
9	300	20	96

[a] *Partial decomposition occurred at higher power*
[b] *0.2 mL of ethanol added*

the first irradiation for 30 s at 300 W the homogeneity of the reaction mixture changes due to formation of small amounts of type **2** salts.

The reaction mixture is then taken out, mixed again for 90 s and then heated at the same power level for additional 30 s (Table 1). This step is repeated until the formation of a colored product. At this stage, the unreacted starting materials are removed by washing with ether and the product dried under vacuum at 40°C.

A series of salts **2a-g** were prepared by microwave heating and the protocol is then compared with similar preparation using conventional heating (room tempeture stirring for iodomethane and heating on the oil bath unfer reflux for the rest alkyl halides). The results are summarized in Table 2.

The **2a-g** salts were initially prepared using a 10% haloalkane molar excess, but the amount was later increased. Due to evaporative loss, the preparation salts **2a-g** in open vessels often requires a large molar excess of haloalkane to obtain good yields. For example, 50% molar excess of 1-bromobutane haw previously been used to prepare *N*-butyl-3-boronopyridinium ester bromide (89% yield). The rate at which the quaternization of pyridine proceeds follows the conventional order, R-I > R-Br > R-Cl [24]. Due to the high reactivity of the iodides excellent yields are obtained in all cases with minimum exposure time.

The progress of the reaction was monitored by ^{1}H NMR (Fig. 3). Further evidence for characterization of synthesized compounds was obtained from the mass spectral data. Furthermore, conversions (based on ^{1}H NMR spectroscopy) of 99.8% for **1** were obtained, making the current synthetic route more cost- and reagent efficient, and hence greener.

An experiment was performed to determine if the reaction time in a polar solvent would be reduced in a microwave oven. A comparison of the heating rate of a mixture of **1** and RX in ethanol with that of mixture of **1** and RX without solvent in an open vessel in the microwave oven at 300 watts, shows that the

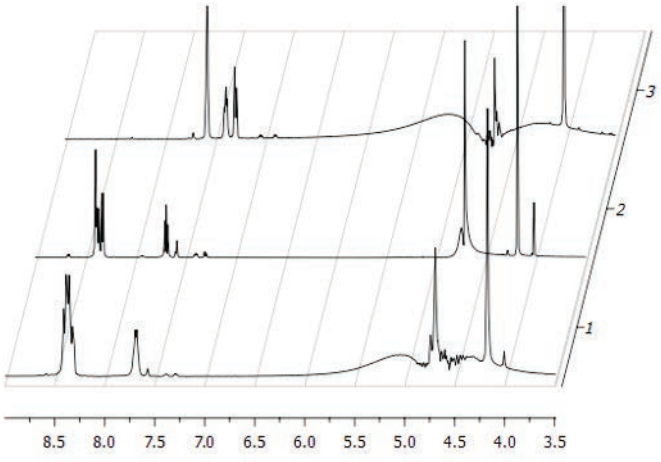

Figure 4. ^{1}H NMR (H_2O-D_2O mixture, 25°C, water suppression) spectra of 3a iodide (0,01 M phosphate buffer solution) in the presence of 10-fold excess of H_2O_2 *vs* time: 2 h (bottom line); 72 h (medium line); 1-methyl-3-hydroxyl pyridinium iodide (upper spectrum).

Table 2. Microwave-assisted preparation of compounds **2a-g**.

Compound	RX	[RX], mmol	MW Power, W	Time, min	Yield % [a]	Yield %[b] (time/days)
2a	CH_3I	4	180	3	94	75 (7)
2b	C_4H_9I	2.5	180	18	82	50 (5)
2c	$C_6H_{11}I$	2.5	180	12	86	75 (7)
2d	$C_8H_{17}I$	2.5	180	25	84	75 (7)
2e	$C_{10}H_{11}Br$	1,25	180	33	76	65 (10
2e	$C_{10}H_{21}I$	1.25	300	18	90	75 (10)
2f	$C_{12}H_{25}Br$	1.25	300	18	75	75 (15)
2f	$C_{12}H_{25}I$	1.25	300	20	96	75 (15)
2g	$C_{16}H_{33}I$	1.25	300	20	96	75 (15)

[a] *Using MW, with 0.2 mL of ethanol.*
[b] *Alternative heating method (oil bath)*

dilute solutions of substrate in ethanol are heated very rapidly. This occurs because only the polar molecules in solution absorb the microwave energy [25], superheating the solvent rapidly.

Aryl boronic acid are known to be unstable in the presence of H_2O_2 [26] and small inorganic nucleophiles [27]. At the same time, 3-pyridine boronic acid is one of the most acidic of the known boronic acids, with a pK_a of ca. 4.0 [28], and possesses the most electrophilic boron atom that can best form and stabilize a hydroxyboronate anion. Unlike most boronic acids, it exists in the anionic tetrahedral species at a pH ranging from slightly acidic to strong alkaline, which may facilitate peroxoacid formation in the presence of hydroperoxide anion under “mild” conditions. If they are stable in a water/H_2O_2 solution, *N*-alkyl derivatives of 3-pyridine boronic acid may be of interest as peroxide activators, particularly as the components of “surfoxidants” [29] for oxidative detoxication of organic sulfides [11]. Thus, the problem of how stable these compounds are against deboronation indicative for most aryl boronic acids is of high interest. Some of amphiphilic boronopyridinium salts were reported recently [10] to be reagents for cross-coupling reactions but the stability of alkaline solutions and an interaction with hydrogen peroxide have not been reported.

^{1}H NMR spectra recorded for system **3a**:H_2O_2 (1:10) indicate (Fig. 4) that in the presence of large excess of H_2O_2 a small amount of deboronaton product 3-hydroxypyridinium methiodide appears (see chemical shifts developing upon a time, Fig. 4). During the first 2 h, ca.3% of starting boronic acid undergoes deboronation with hydrogen peroxide, and after 72h, less than 15% of the initial compound undergoes deboronation. Thus, in the case of **3a**, the rearrangement of peroxoarylboronic acid to the boric acid aryl ester reported for phenylboronic acid [30] proceeds much slowly, and salts of type **3** are stable enough to be investigated as peroxide activators and key components for oxidative systems.

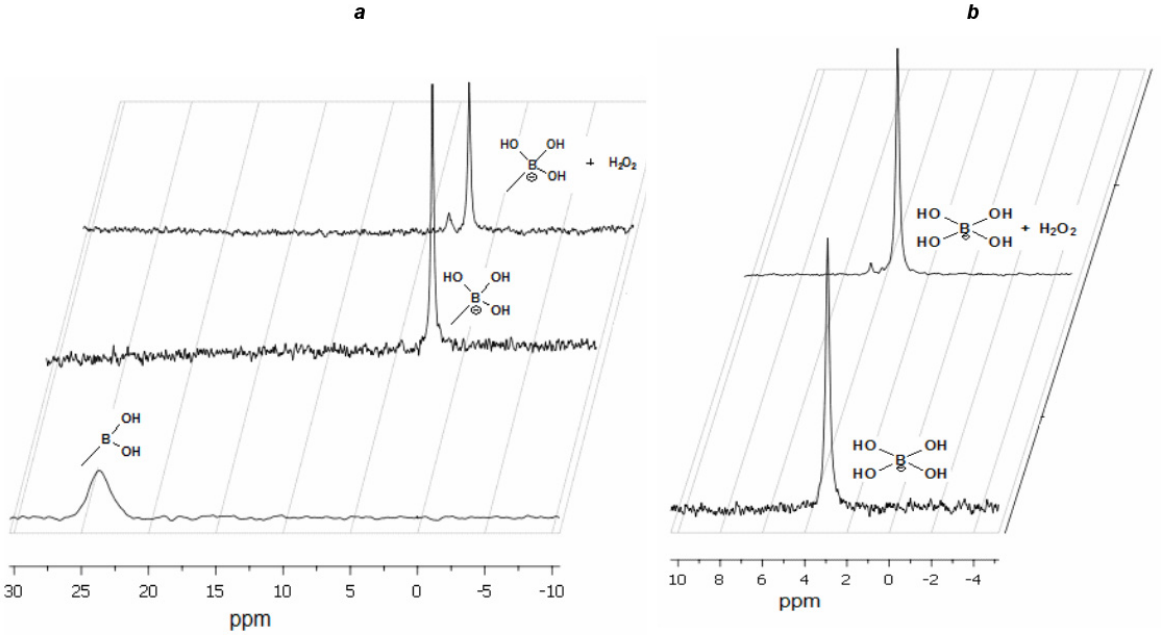

Figure 5. ^{11}B NMR (H_2O-D_2O mixture, BF_3OEt_2 is a reference) spectra of (a) 3a iodide and (b) boric acid in the presence and absence of hydrogen peroxide, pH 10.0; 0.01 M phosphate buffer solution. The bottom spectrum in (a) corresponds to pH 3.3.

^{11}B spectra of salt **3a** and boric acid alone were recorded at different pH's and in the presence of an excess of hydrogen peroxide. All samples were freshly prepared and run within 30 min. At low pH, salt **3a** exist in its trivalent (cationic boronic acid salt) form, characterized by a broad peak with chemical shift at 23.69 ppm (Fig. 5a, bottom spectrum) similarly to that of methylboronic acid reported in [30]. With increasing pH (10.0) the addition of OH to the boronic acid takes place, and tetrahedral (zwitter-ionic boronate form) species appears following by signal shifting to 1.64 ppm (Fig. 5a, medium spectrum). In the case of boric acid, there is a signal corresponding to borate anion at pH 10 (2.94 ppm). The presence of excess of H_2O_2 does not affect main chemical shift drastically (less that 1 ppm towards strong field) but is characterized by appearance of additional peak, with chemical shift of 4.33 ppm and 3.14 ppm for boric acid and **3a**, respectively.

As we can conclude from ^{1}H data, the secondary signal (Fig. 5a) unlikely reflects deboronation that could take place in the presence of peroxide. The new signals are similar for boric and boronic acid and are likely correspond to peroxoacids formation, peroxoboronic (Fig. 5a, upper spectrum) or peroxoboric (**5b**, upper spectra), and is generally in agreement with the investigation of mixture of boric acid and H_2O_2 [31].

4. Conclusions

In conclusion, we have developed a general and rapid method for the synthesis of *N*-alkyl-3-boronopyridinium acid halide salts in nearly quantitative yield, under microwave irradiation. The high efficiency and use of only cosolvent without other additives makes the method useful and attractive for the synthesis of *N*-alkylpyridinium boronic acids. The stability of obtained salts was studied using UV-Vis spectroscopy and ^{11}B, ^{1}H and ^{13}C NMR at different pH, salt concentration, and H_2O_2 content. At 10 times excess of hydrogen peroxides (pH 10.0), less than 15% of degradation products appear after 72 h of exposure. The *N*-alkyl-3-boronopyridinium halides are proposed to be used as peroxide activators and precursors to mild oxidants for the oxidation of organic sulfides.

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